ADAPTATION OF AN X-RAY TUBE FOR TAKING ELECTRON DIFFRACTION PHOTOGRAPHS

KARL A. DAVIS AND JOHN E. EDWARDS
Department of Physics, Ohio University, Athens, Ohio

An x-ray tube having removable target and filament assemblies normally requires considerable auxiliary equipment which is more or less permanently attached to the tube. Such equipment includes an evacuating system, a vacuum gauge, liquid coolant, and a high voltage source. In general, the same type of equipment is required to operate an electron diffraction unit.

In view of this, a demountable x-ray tube in this laboratory was modified to function either as an x-ray tube or as an electron diffraction tube depending upon the attachment of either an x-ray target or an electron diffraction camera.

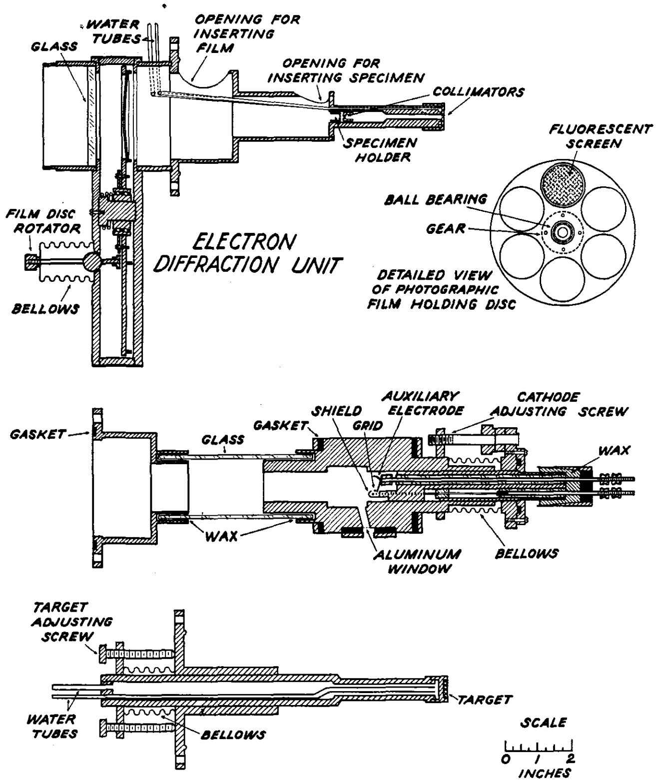

FIGURE 1. Sectional drawing of the combination tube.

THE DUAL PURPOSE TUBE

The basic x-ray tube before modification was designed by Dershem (1936) for use in vacuum spectrographs. The filament assembly and the copper shield surrounding the target and the filament were not changed. The target assembly and the target end of the tube were altered to accommodate the electron diffraction camera.

The drawing at the top of figure 1 shows a sectional view of the electron diffraction assembly which was designed to be inserted into the position normally occupied by the target assembly. The center drawing is a sectional view of the x-ray tube with the target assembly removed. The x-ray target assembly is represented in the lower drawing. The portion of the electron diffraction camera which includes the anode, collimators, and specimen holder, resembles the x-ray target. A brass cap soldered to the end of the hollow stem is pierced in the center by a 0.0135 inch hole which serves as the first collimator for the electron beam. The hollow stem also carries the second collimator and the specimen holder which are easily removed. The second collimator has a diameter of 0.006 inches. The specimen holder and second collimator are adjustable longitudinally. Several by-pass openings in the sample holder prevent breakage of the thin sample during evacuation of the system. Electrons leaving the sample travel a distance of seven inches to the film where a diffraction pattern of maximum diameter 3¼ inches may be formed. The film holding disc holds five films and the fluorescent screen. The disc is held against the front of the camera case by a coil spring but it is easily rotated without disturbing the vacuum. The rotation is accomplished by engaging the larger gear on the disc with a small gear attached to the flexible bellows. By

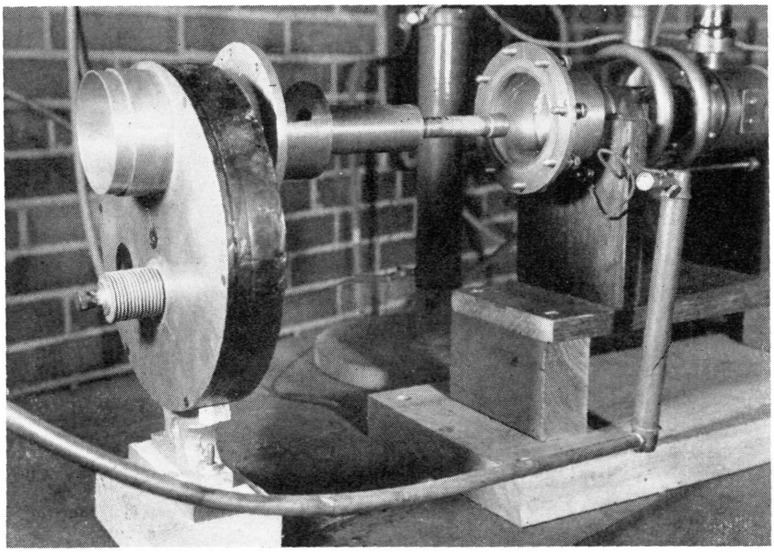

FIGURE 2. Rear view of the electron diffraction camera.

moving the rotator a short distance and re-engaging the gears, a new film can be brought into exposure position. When the fluorescent screen is in exposure position the camera is light tight for the five films, but the fluorescent screen viewing tube is covered by a cap to prevent fogging when a film is moved into position. The films are held in the cells on the disc by springs. Only one rubber ring vacuum seal is involved in converting the tube from x-ray to electron diffraction work. Figure 2 shows a rear view of the camera.

A focused electron beam is supplied by a filament and grid assembly. The electrons are deflected from behind a molybdenum shield by a positive voltage on the grid and auxiliary electrode. They can then come under the influence of the high voltage field. Sharpness of the focus at the first collimator is controlled by adjusting the distance of the cathode assembly from the target. The position of the focal spot can be swept back and forth over the central region of the first collimator by varying the grid voltage. A maximum supply of electrons can thus be obtained by varying the grid voltage and watching the brilliance of the pattern on the fluorescent screen. Grid voltages vary from 50 to 300 volts and are furnished by a small direct current power source. A generous supply of electrons for diffraction photographs can be obtained with target currents less than 0.5 milliamperes.

Voltages as high as 50 kv are supplied to the tube by a full wave rectified source using filter condensers of 0.4 microfarads capacity. The tube is evacuated with a two-stage oil diffusion pump and is frequently operated with the diffraction camera without use of a cold trap on the pump.

PROCEDURE

The tube is being used for instructional purposes in the advanced physics laboratory. A series of five transmission type electron diffraction photographs taken at various voltages can be secured during a two to four hour laboratory period. Samples of gold and silver have been used but the most consistently good results are obtained with MgO. All of the samples are deposited on thin collodion. The collodion film is prepared by dropping a small amount of a collodion-amyl acetate solution on water. After evaporation of the amyl acetate the sample holder is slipped under the film and lifted up slowly, then allowed to dry. Gold or silver may be sputtered on the collodion film but a most satisfactory sample for instructional purposes can be prepared quickly by holding the sample holder in the "smoke" stream about eight inches above a burning magnesium ribbon.

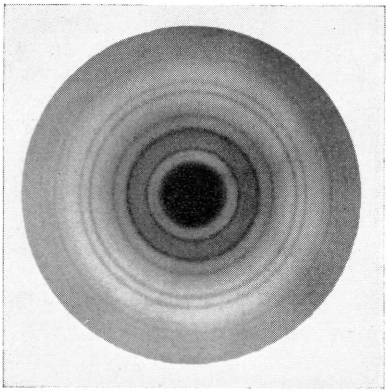

FIGURE 3. Electron diffraction pattern of MgO taken at 26 kv.

After inserting the sample and films in the camera, it is bolted into position and the system evacuated. The diffraction pattern is first checked with the fluorescent screen, then the high voltage is removed, the grid voltage is reversed, and a film is moved into exposure position. The grid provides a very convenient method for making exposures. It is biased negatively to deflect the electron beam from the collimator until the high voltage builds up to a pre-set value. Then the grid is made positive for the desired exposure time. Satisfactory photographs can be obtained with exposure time as low as 0.5 sec. Figure 3 shows a diffraction pattern of MgO taken at 26 kv. The central region of the pattern was over-exposed in printing the photograph to show several circles on one print.

The focusing requirements in securing an adequate supply of electrons are not severe. A central spot can be seen on the fluorescent screen when the tube is operated without grid or filament shield. It is believed that many types of demountable x-ray tubes might be adapted for electron diffraction work. Where more space is available at the target end of the tube provision might easily be made for manipulation of the sample.

LITERATURE CITED

Dershem, E. 1936. A shielded filament x-ray tube for pure x-ray spectra. Rev. Sci. Instr., 7: 86–89.